Characteristics of Prepared Coke–biochar Composite and Its Influence on Reduction of NO_x Emission in Iron Ore Sintering

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Substituting biofuel for coke breeze in the iron ore sintering process has become increasingly attractive. However, coke breeze and straw char separately distributed in the sinter bed lead to poor-quality sinter because of their different combustibilities. In this investigation, a coke–biochar composite (CBC) is produced from coking coal and raw straw, and its behaviour when used as a fuel for sintering is investigated. The results show that the coke and straw char in the CBC interpenetrate and adhere together. Compared with separately distributed coke breeze and straw char, this structure effectively restrains separate combustion of the CBC components. A CBC with 40–60% straw char is recommended as a replacement for coke breeze, with only small changes in sinter quality. The fuel nitrogen conversion rate is decreased by 8.3% during CBC combustion, possibly owing to reducing reactions between C (CO) and NO_x, which further decrease NO_x emission.

KEY WORDS: agglomeration; iron ore sintering; bio-energy; coke–biochar composite; NO_x emission reduction.

1. Introduction

Iron ore sinter is the chief iron-bearing raw material for blast furnaces in integrated steel works in China. However, the sintering process typically consumes 9–12% of the total energy of steel production, which is typically provided by the combustion of solid fossil fuels such as coke.^{1,2)} It has been shown that diverse pollutants such as CO₂, SO_x and NO_x are produced by the combustion of fossil fuels.^{3,4)} In particular, more than 90% of NO_x comes from this source.⁵⁾ To address this problem, the behaviour of biomass fuels when used in the sintering process has attracted attention. The use of charcoal in sintering was investigated by the Australian CSIRO,^{6–8)} and the behaviour of uncarbonised biomass fuels such as olive residues and sunflower husk pellets in the sintering process was examined by researchers at Corus.^{9,10)} Their findings showed that the use of biomass fuels led to a reduction in SO_x and NO_x emissions because of their lower sulphur and nitrogen contents. The emissions of CO₂ from biomass fuels were nearly zero as they were carbon-neutral. Since China has the world's largest capacity for sinter production, there is an urgent and increasing need for supplemental clean fuels.

China has an abundance of straw resources, with 17.27% of the world's total amount, representing an annual output of 800 million tons. If fully developed, this could be used as an energy equivalent of 300 million tons of coal.¹¹⁾ However, the availability is less than 45% due to open burning and abandoning.^{12,13)} Therefore, converting straw into an

alternative fuel to coke breeze is both environmentally and economically attractive.

Before putting biomass fuels into use on an industrial scale, our group investigated the properties and sintering behaviour of charcoal, charred straw, *etc.*^{14,15)} Considerable benefits of these biomass fuels were observed with regard to reduced emissions of CO₂, SO_x and NO_x. However, they were found to exhibit greater porosity and specific surface area than coke, especially in the case of straw char, which resulted in degradation of sinter quality if an excessive proportion of coke breeze was replaced. In the present investigation, a novel approach is proposed for increasing the adaptability of straw char during the sintering process, including combining coke with straw char in a coke–biochar composite (CBC). The characteristics of this CBC are examined, and a laboratory-scale sinter pot is used to evaluate its effect on sinter production.

2. Materials and Methods

2.1. Material Properties

In sinter production, iron ores, fluxes (dolomite, limestone and quicklime), solid fuels and return fines (with a sinter of –5 mm) are indispensable raw materials. In our study, they were all provided by an integrated steelworks in China. Among them, mixed iron ores were mainly used to regulate the contents of Fe and SiO₂ in the sinter, while fluxes were used to adjust the basicity (CaO/SiO₂) and MgO content. According to the scheme adopted in the sinter plant, the Fe, SiO₂ and MgO contents and the basicity were kept at 56.38%, 4.92%, 2% and 1.9, respectively, in the finished sinter cake. The chemical composition and proportions of

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Table 1. Chemical composition of raw materials and their proportion in the mixture (mass%).

Raw material	Chemical composition							Proportion
	TFe	SiO ₂	CaO	MgO	Al ₂ O ₃	FeO	LOI ^a	
Mixed iron ores	60.74	4.65	1.9	1.54	1.92	9.24	2.64	64.93
Dolomite	0.21	0.87	31.57	19.68	0.22	0.13	46.91	0.53
Limestone	0.14	1.31	50.19	3.30	0.32	0.10	42.27	3.05
Quicklime	0.40	2.85	77.92	3.64	0.75	0.23	11.29	4.56
Return fines	56.38	4.92	9.36	2.00	2.07	8.58	0.00	23.08 ^b
Fuel	—	—	—	—	—	—	—	3.85

^aLOI=loss on ignition; ^bThe proportion of return fines will be changed when experiments are performed to determine the return fine balance

Table 2. Proximate and ultimate analyses of fuels (dry basis).

Fuel	Ultimate analyses (mass%)					Proximate analyses (mass%)			Calorific value (MJ/kg)
	C	H	O	N	S	Ash	Volatiles	Fixed carbon	
Coke breeze	81.84	2.46	1.03	0.72	0.224	19.54	5.88	74.68	27.06
Raw straw	45.44	5.33	46.96	0.25	0.078	5.10	76.15	18.75	18.80
Soft coal	76.94	4.08	16.01	1.09	1.600	11.14	28.79	59.07	22.50

individual raw materials are given in **Table 1**. The influences of different fuels on the elemental constituents of the mixture depend mainly on the ash composition, and this influence was neglected in our investigation for lower fuel ratio in mixtures and lower ash ratio in fuels.

Coke breeze, raw corn straw and soft coal were employed in this investigation. Of these, the raw straw and soft coal served as raw materials for preparing the straw char and the CBC. The coke breeze and soft coal were provided by a sinter plant and a coking plant, respectively. Most of the coke breeze was in the size range 0–3 mm. The raw straw was provided by a state-owned bio-energy corporation. Ultimate analyses were performed using a CHNOS Elemental Analyser, model Vario EL (Elementar, Germany), while proximate analyses were performed with an Automatic Proximate Analyser, model SDTGA5000 (Sundy, China). Results are given in **Table 2**, where it can be seen that the raw straw consisted mainly of C, H and O. Its volatiles content was greater than 76%, while its fixed carbon content was only about 18%, and therefore it was assumed to have been pretreated before use. The soft coal was a typical kind of fat coal, with higher volatile content. It provided sufficiently cohesive plastic mass to allow the combination of separate particles during the coking process.¹⁶⁾

2.2. Experimental Methods

2.2.1. Fuel Preparation and Property Examination

The straw char and CBC were prepared in an electrically heated shaft furnace. The unit is depicted schematically in **Fig. 1**. Before carbonising, raw straw and soft coal crushed to –2 mm were moulded into pellets 10 mm long and 10 mm in diameter by a pelletiser without any binder at room temperature. The pellets were produced at a maximum pressure of 180 MPa, which was maintained for 1 min. For carbonisation, briquetted pellets were first charged into a quartz reactor, which was then moved into a furnace. The

Fig. 1. Schematic diagram of carbonisation equipment.

pellets then underwent a two-stage carbonisation process. In the first stage, they were heated to 500°C at a rate of 25°C min^{−1} for a residence time of 30 min. They were then heated to 700°C with the same rate and residence time. The whole process was conducted under an inert atmosphere with an argon flow of 0.1 L min^{−1}. After carbonisation, the straw char was cooled to room temperature in argon with a flow rate of 0.1 L min^{−1}, and its weight was then measured. The percentage yield of carbonised fuel was calculated as

$$W = M_1/M_2 \times 100\% \quad (1)$$

where M_1 is the mass of final solid product (g) and M_2 is the mass of dry straw and soft coal charged for carbonisation (g). An optical microscope, model DMRXP (Leica, Germany), was used to analyse the microstructure of fuel samples.

The combustibilities of the coke breeze, straw char and CBC were examined using a simultaneous TG-DSC analyser, model STA449C (Netzsch, Germany). From

Fig. 2. Schematic diagram of laboratory sinter pot.

these analyses, differential scanning calorimetry (DSC), derivative thermogravimetry (DTG) and thermogravimetry (TG) curves were obtained. Before analysis, 10 ± 0.1 mg samples with a size range of 0.125–0.25 mm were accurately weighed and then charged into an Al_2O_3 crucible. The crucible was placed in a sample holder inside the furnace of the analyser, and the analyses were conducted under the same conditions using appropriate baseline correction. The samples were heated to $1\,000^\circ\text{C}$ to achieve complete combustion. Throughout the process, the heating and airflow rates were kept at $10^\circ\text{C min}^{-1}$ and 0.1 L min^{-1} , respectively. Combustion parameters, including the peak values $V_{\max}$ and $Q_{\max}$ on the DTG and DSC curves and the initial burning temperature T_i and the ending temperature T_e on the DTG curves, were determined. Here, $V_{\max}$ and $Q_{\max}$ are respectively the maximum weight loss rate of the fuel and the maximum value of the heat release value, which are related in a positive manner to the reactivity of the fuel.¹⁷⁾ T_i is the temperature at which the weight loss rate first reaches $1\% \text{ min}^{-1}$ on the DTG curve after the moisture evaporation stage and T_e is the temperature at which the weight loss rate reaches $1\% \text{ min}^{-1}$ on the final part of the DTG curve.¹⁸⁾

2.2.2. Description of Sintering Trials

Sintering trials were conducted in a sinter pot of depth 700 mm and diameter 180 mm, a schematic diagram of which is shown in Fig. 2. The raw materials were blended in given proportions, with water being added to provide the prescribed moisture content. The mixture was then charged into an electrically powered drum of depth 1 400 mm and diameter 600 mm for granulation, which was conducted at 15 rev min^{-1} for 4 min. A sample was taken from the granulated mixture, and its permeability was measured as the pressure drop across a green bed of depth 200 mm and diameter 100 mm at an air flow rate of $10 \text{ m}^3 \text{ h}^{-1}$. This air flow rate was chosen as an approximation to the superficial gas velocity during practical sinter trials.

A 1 kg sinter of size range 10–16 mm was charged into the sinter pot as a hearth layer, and the granulated mixture was then fed in. After feeding, the fuel in the surface layer was initially ignited by an ignition hood, and the combustion front then moved downwards with the support of a downdraught system. The ignition period was 1 min at $1\,050 \pm 50^\circ\text{C}$, with a pressure drop of 5 kPa. However, the

pressure drop was changed to 10 kPa for sintering and then to 5 kPa for cooling. The total sintering time and total flue gas flow were measured from the start of ignition to the point at which the sinter waste gas had reached its maximum temperature. A thermocouple was installed 200 mm beneath the bed to monitor temperature changes. Two types of infrared flue gas analyser, a DELTA 65-3 and a Vario Plus (MRU Corporation, Germany), were used to analyse the concentration of NO_x in the flue gas.

The return fine balance (RFB), which is the ratio of the mass of produced return fines to the mass of afforded return fines, was taken as an important index to validate the tests of sintering speed, sinter yield, tumble index, productivity, etc., and these tests were conducted only when the RFB was in the range of 1 ± 0.05 . The sintering speed is the ratio of layer height and sintering time. The sinter yield is the percentage of sinter above 5 mm after screening, with the mass of the hearth layer subtracted. The productivity is a measure of the quantity of sinter produced per unit area and unit time. The tumble index is a measure of sinter strength, based on the methodology outlined in ISO3271(2007).

3. Results and Discussion

3.1. Influence of the Replacement of Coke Breeze by Straw Char on Sintering Indices

The weight of straw char for each run was calculated on the basis of heat balance, such that the straw char provided an equal amount of heat to the coke breeze that it replaced. The straw char had previously been crushed and screened, and only the portion with the same size range as the coke breeze was selected. The appropriate moisture content of raw mixture for each run was determined by reaching desirable permeability, sinter yield and tumble index. Table 3 shows the results of the sintering tests.

As can be seen from Table 3, the mixture permeability, which was a critical factor determining the sintering speed, remained stable for different runs. However, the sintering speed still increased with increasing proportion of straw char. In particular, for 40% straw char, the sintering speed was much higher than in the absence of straw char. The main reason for this increase in sintering speed was the marked difference in combustibility exhibited by straw char in comparison with coke breeze, which can be clearly

Table 3. Effects of straw char replacing coke breeze on sintering indices.

Replacement ratio (mass%)	Moisture content/%	Sintering speed (mm · min ⁻¹)	Sinter yield (mass%)	Tumble index (mass%)	Productivity (t · m ⁻² · h ⁻¹)	Mixture Permeability (^a JPU)	^b HBT ₂₀₀ (°C)
0	7.25	22.01	73.30	65.32	1.51	38.50	1 273
20	7.50	23.50	68.79	64.80	1.45	38.91	1 210
40	7.75	25.35	63.05	61.15	1.25	39.13	1 185

^aJPU = Japanese Permeability Unit; ^bHBT₂₀₀=highest bed temperature at 200 mm.

Fig. 3. Combustion properties of different fuels. $\Delta T = T_e - T_i$; (a) Coke breeze; (b) Straw char.

observed in **Fig. 3**. Straw char was more reactive than coke breeze owing to its higher $V_{\max}$ and $Q_{\max}$ values. Its initial temperature was merely 351.4°C, while that for coke breeze was 513.6°C. The different ΔT values also indicated that the duration of combustion of straw char was shorter than that of coke breeze.

Physically blended coke breeze and straw char are distributed separately in the sinter bed. Because of their different combustibilities, the straw char will combust before the coke breeze, and because of their different initial temperatures and burning speeds, they will not release heat in a similar manner. This makes it difficult to achieve the temperature and duration needed to form sufficient liquid calcium ferrite to facilitate cohesion during the condensation–crystallisation stage of the sintering process,^{19,20)} with the result that the formation of strong linkages among individual ore particles will be impeded. The decrease in highest bed temperature at 200 mm (HBT₂₀₀) with increasing proportion of straw char shown in Table 3 confirms the effects of the different combustibilities of the straw char and coke breeze. As a result, increasing the proportion of straw char led to decreases in the sinter yield and the tumble index of the sinter. In particular, when the proportion exceeded 20%, apparent decreases were observed.

3.2. Characteristics of CBC

Since the combustibilities of straw char and coke breeze do not match, they undergo combustion separately in the sinter bed, leading to changes in sinter quality. Therefore, in view of the potential interaction between straw char and coke, a CBC consisting of closely combined straw char and coke was prepared. To determine the relative proportions of straw char and coke in the CBC, a number of trials were conducted. The yield and calorific value for coke and straw

char when carbonised separately were analysed, with the results shown in **Table 4**. The proportion of straw char in the CBC was determined on the basis of heat balance, with the relationship between the masses of soft coal and straw for preparing the CBC being given by

$$m_1 = \frac{r}{1-r} \frac{Y_2}{Y_1} \frac{C_2}{C_1} m_2 \quad \dots\dots\dots (2)$$

where m_1 is the mass of soft coal, m_2 is the mass of raw straw, r is the ratio of coke in the CBC, Y_1 is the yield of coke, Y_2 is the yield of straw char, C_1 is the calorific value of the coke and C_2 is the calorific value of the straw char. CBCs with different ratios of straw char were prepared. As shown in Table 4, owing to the differences in various properties between soft coal and raw straw, increasing the proportion of straw char in the CBC led to a marked drop in yield. With increasing proportion of straw char, the level of fixed carbon and the calorific value of the CBC also decreased slightly, there was a gradual increase in the content of volatile matter, and, because of the lower nitrogen content of straw char, there was a marked reduction in nitrogen content.

It can be seen from the images of CBC microstructure in **Fig. 4** that there was mutual adherence and interpenetration of its two components, with this interaction becoming more pronounced as the proportion of straw char increased.

The combustibility properties of the different fuels are shown in **Fig. 5**. For physically blended straw char and coke breeze, the DSC and DTG curves show double peaks, with the first peak indicating principally the combustion of straw char as its lower initial temperature and the second peak the combustion of coke breeze. In contrast, instead of combusting in separate stages, the straw char and coke in the CBC exhibited integrated combustibility, which is apparent from

Table 4. Yield and proximate analyses of coke, straw char and CBC (dry basis).

Raw material	Product fuel	Yield (mass%)	Ash (mass%)	Volatile matter (mass%)	Fixed carbon (mass%)	Calorific value (MJ/kg)	N (mass%)
Soft coal	Coke	74.40	15.08	4.31	80.61	29.30	0.75
Straw	Straw char	24.95	20.30	4.52	75.18	27.56	0.18
Soft coal+ Straw	CBC _{20%} ^a	52.71	16.25	4.35	79.40	28.85	0.65
	CBC _{40%}	40.79	17.30	4.38	78.32	28.50	0.53
	CBC _{60%}	33.62	18.17	4.46	77.37	28.20	0.42
	CBC _{80%}	28.50	19.25	4.51	76.24	27.75	0.26

^aCBC_{20%}, 40%, 60%, 80% = CBC containing 20%, 40%, 60%, 80% straw char by calculating.

Fig. 4. Microstructure of CBC containing different proportions of straw char. (a) Coke; (b) CBC with 20% straw char; (c) CBC with 40% straw char; (d) CBC with 60% straw char; (e) CBC with 80% straw char; (f) Straw char.

Fig. 5. Combustibilities of different fuels. (a) 40% straw char + 60% coke breeze; (b) CBC with 40% straw char.

the shapes of the TG, DTG and DSC curves. The high reactivity of straw char was suppressed to some extent, probably because of the presence of coke nearby. The initial temperature for the CBC was 482.6°C, which was close to that

of coke breeze and much higher than that of the physically blended fuels. The CBC also showed a similar ΔT to that of coke breeze. Both the $Q_{\max}$ and the $V_{\max}$ of CBC showed only a small difference to that of coke breeze.

3.3. Application of CBC in the Iron Ore Sintering Process

3.3.1. Improvement in Sintering Quality

To investigate the effects on sintering indices, CBCs containing 40%, 60% and 80% straw char were selected as replacements for coke breeze. They underwent pretreatment before use, with samples having a similar size distribution to that of coke breeze being selected. The amounts of CBC to be used as complete replacements for the coke breeze were determined on the basis of heat balance.

As shown schematically in **Fig. 6**, in contrast to physically blended coke breeze and straw char, the use of CBC avoided the separate distribution of coke and straw char in the sinter bed and thus contributed to restricting their separate combustion. As a result, heat was released by the fuels in a uniform and intense manner, which helped to achieve the temperature and duration required to form enough calcium ferrite in the bed.

As can be seen from **Table 5**, when CBC with 40–60% straw char was used, the sintering speed was increased compared with using coke breeze alone, although for 40% straw char, the increase was only slight. The sinter yield and tumble index also changed slightly. The improved yield and tumble strength were closely linked to the changes in sinter microstructure (**Fig. 7**). When physically blended straw char and coke breeze were used, calcium ferrite generated in the sinter bed failed to effectively coat the individual ore particles and bind them together (**Fig. 7(a)**), with the result that a greater number of pores appeared inside the sinter. With the use of CBC, the HBT₂₀₀ was maintained at a similar level to that when coke breeze was used. Strong links were formed between ore particles with the help of evenly distributed calcium ferrite (**Fig. 7(b)**).

When the ratio of straw char exceeded 60%, there was a marked increase in sintering speed, but the product ratio, tumbler index and productivity decreased significantly.

From these results, it appears that CBC with 40–60% straw char should be recommended to replace coke breeze for sintering of iron ore.

Fig. 6. Comparison of fuel distributions in sinter bed. (a) Physically blended straw char and coke breeze; (b) CBC.

3.3.2. Reduction in NO_x Emission

The use of CBC was compared with that of physically blended straw char and coke breeze in terms of their contribution to reduction of NO_x emission. The relative contributions of physically blended straw char and straw char in CBC to reduction of NO_x emission were assessed by the fuel-N conversion rate R , which stood for the ratio of fuel-N to NO_x, given by

$$R = \left(\frac{Q \int_0^{t_s} c_{t1} dt}{\sum w_N m} \frac{M_N}{M_N + M_O} + \frac{Q \int_0^{t_s} c_{t2} dt}{\sum w_N m} \frac{M_N}{M_N + 2M_O} \right) \cdot 100\% \quad (3)$$

where Q is the sampled total flue gas flow, t_s is the total sintering time, c_{t1} is the mass concentration of NO at time t , c_{t2} is the mass concentration of NO₂ at time t , w_N is the nitrogen content of straw char or coke, m is the mass of straw char or coke used during sintering, M_N is the molecular weight of nitrogen and M_O is the molecular weight of oxygen.

As can be seen from **Fig. 8**, the concentration of NO was far higher than that of NO₂, which indicated that NO_x emitted during the sintering process was mainly in the form of NO. When straw char and CBC were used, the emission concentration of NO_x was reduced because of the lower nitrogen content of straw char. The calculated results indicated that nitrogen conversion rates for coke breeze, physically blended straw char and coke breeze, and CBC were 62.4%, 61.1%, and 52.8%, respectively. Compared with physically blended straw char and coke breeze, CBC reduced the nitrogen conversion rate by 8.3%, leading to a further reduction in NO_x emission. A possible mechanism for the degradation of NO_x by CBC is shown in **Fig. 9**. Because of the interpenetration of the straw char and coke components in CBC, the straw char, with its higher reactivity, was able to generate more CO, which diffused to nearby coke particles, around which a reducing atmosphere thus formed. As the coke had a higher nitrogen level, NO_x was easily generated at high temperature and then diffused

Fig. 7. Influence of different fuels on sinter microstructure. (a) Physically blended 40% straw char + 60% coke breeze; (b) CBC with 40% straw char + 60% coke.

Table 5. Effects of CBC on sintering indices.

Fuel types	Proportion of straw char (mass%)	Fuel ratio (mass%)	Moisture content (mass%)	Sintering speed (mm · min ⁻¹)	Sinter yield (mass%)	Tumble index, (mass%)	Productivity (t · m ⁻² · h ⁻¹)	Mixture permeability (JPU)	HBT ₂₀₀ (°C)
Coke breeze	0	3.50	7.25	22.01	73.30	65.32	1.51	38.50	1 273
	40	3.30	7.50	22.25	72.18	65.01	1.50	38.12	1 270
CBC	60	3.36	7.50	23.12	70.05	64.87	1.48	38.91	1 251
	80	3.42	7.75	25.30	66.50	61.30	1.15	37.88	1 195

Fig. 8. Emission concentrations of NO_x . (a) Emission concentration of NO ; (b) Emission concentration of NO_2 .

Fig. 9. Potential mechanism of reduction in NO_x emission.

to the straw char. During diffusion, potential reduction reactions (a), (b) and (c) suppressed the conversion of fuel nitrogen into NO_x . Therefore, the reduction in emission of NO_x with the use of CBC was due not only to the decrease in nitrogen levels with the addition of straw char. However, more research is needed to determine the exact mechanism underlying the decreased nitrogen conversion rate for CBC.

4. Conclusions

An investigation has been performed into the preparation of CBC and its application as an alternative to coke breeze as a fuel in iron ore sintering. The following conclusions can be drawn:

(1) Compared with coke breeze, straw char is characterised by a lower ignition temperature and a shorter burning duration. Because of the different combustibilities of coke breeze and straw char, there is a considerable degradation in sinter quality when more than 20% of coke breeze is replaced by straw char.

(2) In the prepared CBC, there is close binding and interpenetration between the straw char and coke. This structure effectively suppresses separate combustion of these two components.

(3) CBC with 40–60% straw char is recommended as an

alternative to coke breeze as a fuel in sinter making, with the sinter quality being only slightly different from that obtained with coke breeze alone.

(4) For CBC with 40% straw char, the nitrogen conversion rate is reduced by 8.3% compared with that for the same proportion of straw char physically blended with coke breeze. Degradative reactions between C (CO) and NO_x may explain the further reduction in NO_x emission.

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